

Air Monitoring for Methyl Isothiocyanate During a Sprinkler Application of Metam-sodium

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June 1994

ENVIRONMENTAL HAZARDS ASSESSMENT PROGRAM

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EH 94-02

ABSTRACT

In response to complaints of odor and irritation from metam-sodium applications, this study was conducted to determine possible air concentrations of methyl isothiocyanate (MITC), the major breakdown product and active fumigant of metam-sodium, during a "worst case" application scenario. Metam-sodium was applied by a fixed-set sprinkler system to a 20-acre fallow field in Kern County in August, 1993.

The Environmental Hazards Assessment Program of the Department of Pesticide Regulation (DPR) collected air samples during application and the following 68 hours at 10 locations around the field, ranging from 5 m to 150 m from the edge of the application area. Hydrogen sulfide and carbon disulfide levels, minor breakdown products of metam-sodium, were also monitored during several periods following application.

Maximum MITC concentrations occurred during the application period where downwind levels measured 2450 ppb at 5 m from the field edge and 1320 ppb at 150 m from the field edge. The next highest levels occurred during the interval following the watering-in period. Hydrogen sulfide readings were highest during application, ranging from below the detection limit to 76 ppb at a distance of 75 m from the field. All samples analyzed for carbon disulfide were below the laboratory quantification limit of 4 ppb.

Acknowledgements

The authors would like to extend their appreciation to personnel of the Department of Pesticide Regulation and Department of Food and Agriculture for all their efforts in sample collection and chemical analysis. We would also like to extend a hearty thank you to the personnel at Crop Production Services in Bakersfield, Calif., and Zeneca Ag Products. Special thanks to the grower, without whom this study would have been impossible and a lot less enjoyable.

Disclaimer

The mention of commercial products, their source or use in connection with material reported herein, is not to be construed as either an actual or implied endorsement of such product.

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INTRODUCTION

Metam-sodium (sodiumN-methyldithiocarbamate; CAS 137-42-8) is a general soil sterilant used to control nematodes, weeds, and soil fungi. It is a water soluble liquid (722 g/l at 20° C) that can be directly injected into the soil or metered into a sprinkler irrigation system (chemigation) as a preplant incorporated biocide to prepare fallow fields for planting. As a widely used chemical in California, metam-sodium was applied at 2,691,654 kg and 2,216,842 kg of active ingredient (ai) in 1990 and 1991, respectively (CDPR 1990, CDPR 1991).

Metam-sodium breaks down primarily into methyl isothiocyanate (MITC) which functions as the principal pesticidal agent. Metam-sodium is rapidly transformed in soil with a reported half life of < 30 min to 4 hours (Smelt and Leistra 1974, Gerstl et al. 1977a,b). Almost complete transformation has been observed in 3 hours at 12 °C in moist loamy soil, and from 3 to 4 hours at 21 °C, and up to 6 hours at 12 °C in moist humic sandy soil (Smelt and Leistra 1974). Although the transformation rate is slightly higher during illumination, Burnett and Tambling (1986) found that soil residues during both sunlight and dark conditions are reduced to 5% of the applied metam-sodium within 2 hours. In laboratory experiments, Smelt et al. (1989) tested soils ranging from sandy to loamy textures and found that more than 90% of metam-sodium was transformed to MITC in all of the soils. The fastest conversion rates were found in the loamy soil, with the maximum MITC concentrations measured 2 hrs after application. In the other humic and sandy soils, with lower pH, the maximum concentrations of MITC were measured 7 hrs and 24 hours, respectively, after application of metam-sodium.

There are other metam-sodium degradates produced depending on the existing environmental conditions. At pH 5, the major products of hydrolytic degradation are methylamine, MITC, and carbon disulfide (CS₂), and minor hydrolytic degradation products include elemental sulfur and 1,3-dimethylthiourea. In aqueous pH 7 solutions at

25 °C, MITC, N-methylthioformamide, methylamine, and elemental sulfur are major products from photolytic degradation, and minor photolytic degradation products include n-methylformamide, CS₂, carbon oxide sulfide, and hydrogen sulfide (H₂S) (Chang and Myers 1986).

Urban encroachment on agricultural lands has raised concerns of residents to off-site movement of the breakdown products of metam-sodium following field application. Recently, several complaints have been filed with county agricultural commissioners (CDPR 1993) from persons living or working adjacent (≤ 0.8 km) to metam-sodium treated fields. Regardless of which application method (chemigation or injection) was used, reported symptoms included eye irritation, nausea, and headaches.

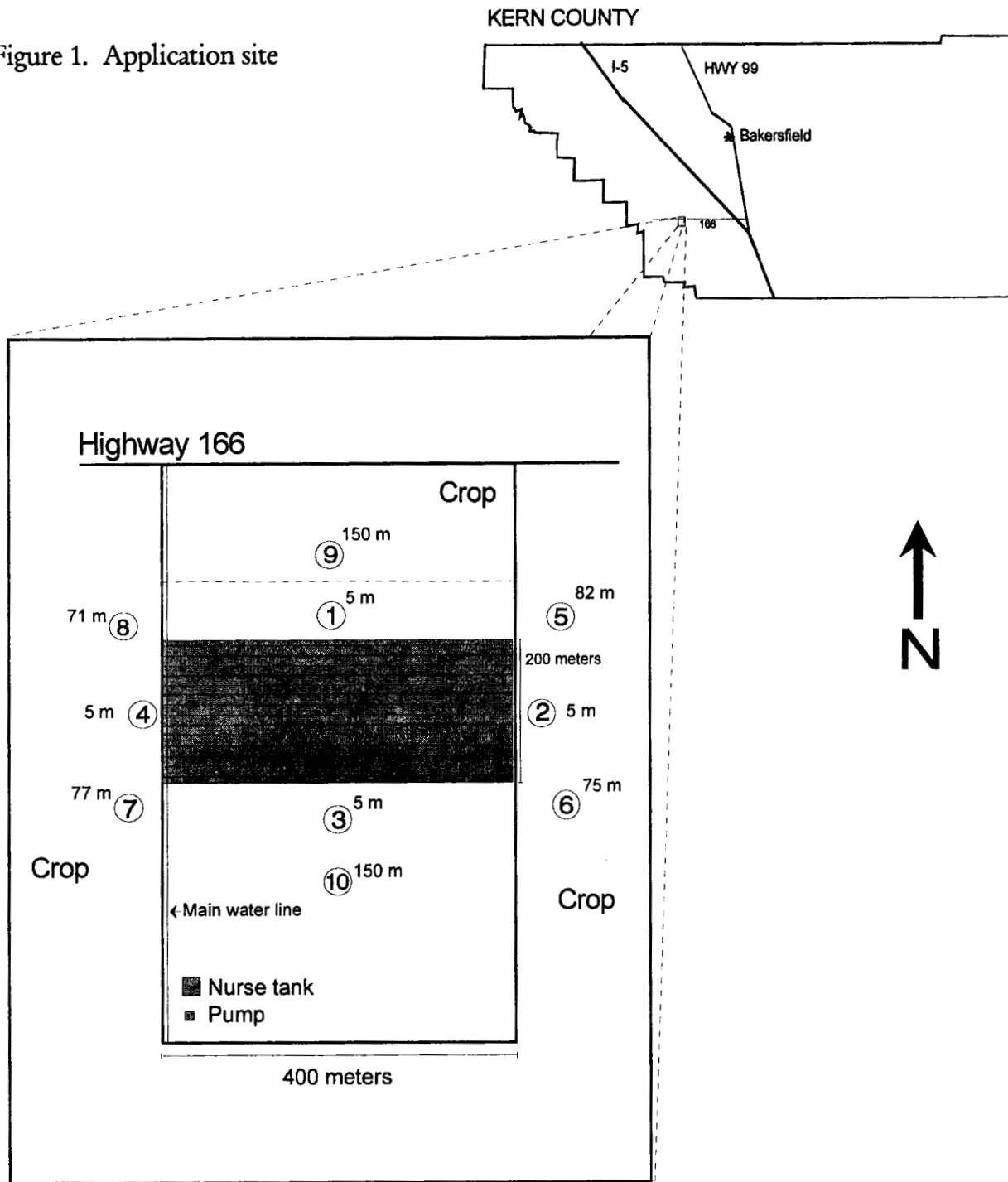
The objective of this study is to monitor MITC, H₂S, and CS₂ air concentrations during a field application of metam-sodium.

MATERIALS AND METHODS

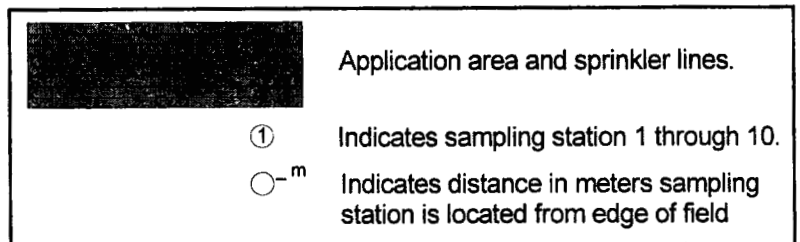
Application Site

Application was to be a worst case scenario in which chemigation occurred during high air temperature, low humidity, and warm soil temperatures at the highest allowable application rate of 935 l/ha (100 gals/acre). Kern County was selected as the target area because of high use of sprinkler applied metam-sodium during the summer months. The site selected for the study was located approximately 32-km south of Bakersfield. The 7.7-ha site was located within a 32-ha field surrounded by cotton and bean fields. Two weeks prior to the application, a bell pepper crop was disced under and the field was left fallow. Fourteen fixed-set sprinkler lines were set east-west across the field, 13.7 m apart, perpendicular to the main line from a pump located 0.4 km south of the site. Sprinkler heads were spaced every 9 m apart on each line for a total sprinkler swath of 400 m by 200 m (Figure 1).

Figure 1. Application site



Legend



During the afternoon before the application, water was run through the sprinklers for a couple hours to pre-irrigate the field and check and adjust the sprinkler system for leaky connections and faulty nozzles. Immediately before the metam-sodium injection, irrigation water was run through the sprinkler system for 1 hour to recheck the system and apply more water to the soil.

Weather data

Wind direction, wind speed, ambient air temperature, and relative humidity were measured during the study with Met-One® sensors on a weather station located at the southwest corner of the treated site. All measurements were averaged and recorded on a data logger (Campbell Scientific, Inc. R-X) for each 1 minute interval, except for wind direction which was taken as an instantaneous measurement once every minute.

Application

Label information prohibits application of metam-sodium when air temperatures and soil temperatures at a 3-inch depth are above 32 °C. The application site must be pre-irrigated to the depth of the metam-sodium treatment to ensure rapid infiltration, and monitored during application and periodically for the following 24 hours for any presence of unpleasant odor. If an odor is present, a water seal must be applied to confine the odor to the soil.

The injection of metam-sodium into the sprinkler system was initiated on August 3, 1993, at 19:40. The metam-sodium solution (Vapam®, ICI) was applied at the maximum label rate of 935 l/ha from a supply tank located 0.4 km south of the treated site at an injection rate of approximately 1200 l of metam-sodium per hour. Irrigation water was pumped through the sprinklers at a rate of 5680 l/min. The application continued for a total of 6 hrs. Immediately after the application, only water was run through the sprinkler system for 1.5 hrs to flush the sprinkler line and incorporate the metam-sodium into the soil leaving a water seal at the surface.

Sampling

Air samples of MITC and CS₂ were taken with two-stage (200/400 mg) coconut charcoal vapor collection tubes (SKC) mounted to personal SKC sample pumps (model 224PCXR7) set at a flow rate of approximately 250 mL/min. Ten sampling sites (Figure 1) were located off the perimeter of the treated area at 3 approximate distances: 5 m, 75 m, and 150 m. The sample tubes were positioned approximately 1.2 m above ground level on metal stakes, except for the sample tubes (4, 7 and 8) located on the western side of the treated site. These samplers were placed at a height of 1.8 m to reduce any interference from the 1.5 m tall cotton plants. No samples were collected at site 9 after the seventh interval due to sprinkler irrigation of the adjacent bean crop. This sampler pump was removed to prevent damage.

The flow rates were checked during each sampling interval with a flow meter calibrated at the EHAP laboratory 2 weeks prior to the study. Since high humidities were expected during the application and watering-in periods, 260/520 mg silica gel tubes (SKC) were mounted in front of the charcoal tubes to remove moisture as recommended in Stauffer Chemical Co. method No. RRC-82-35 for conditions of relative humidities over 80% (Leung 1982). Sample tubes were tightly capped immediately following removal from the air pump and sealed in a plastic bag. They were placed immediately on dry ice and kept frozen until delivery to the laboratory.

Air samples were taken during application, watering-in, and followed by three consecutive 6-hour and four consecutive 12-hour sampling intervals (Table 1). Two 12-hour air samples were collected before application to identify any background levels of MITC. One or two randomly located replicate samples were collected alongside a primary sample during each interval for quality control. Carbon disulfide samples were collected during intervals 1, 2, 3, and 5 at the four sites located 5 m from the field edge. Hydrogen sulfide levels were monitored using an Arizona Instrument Corporation Jerome 621 Hydrogen sulfide ana-

lyzer (minimum detection limit = 3 ppb). This instrument provided instantaneous readings in parts per billion (ppb).

Table 1. Approximate timing of sampling periods.

Sampling Interval	Starting time	Run Time(hrs)
1 (application)	7:30 pm	6
2 (watering in)	1:30 am	1.5
3	3:00 am	6
4	9:00 am	6
5	3:00 pm	6
6	9:00 pm	12
7	9:00 am	12
8	9:00 pm	12
9	9:00 am	12

To test for degradation of samples during field storage, three sample tubes spiked with 5 μ g of metam-sodium were placed in a separate ice chest with dry ice and maintained in conditions similar to the samples. The spiked samples were held on dry ice for 24 days before extraction.

Chemical Analysis

The MITC samples were analyzed by the California Department of Food and Agriculture (CDFA) Chemistry Laboratory Services. MITC was desorbed from each segment of the sample tube in 5 ml of a 0.1% carbon disulfide in ethyl acetate solvent by occasionally agitating for 30 minutes. The extracts were analyzed on a Varian 3700 gas chromatograph equipped with TSD and a HP-FFHAP 10 m x 0.53 mm x 1.0 μ m column. The carrier gas (helium) flow rate was set at 10 ml/min. Column temperature was 45 °C for 3 min and was increased at a rate of 30 °C/min to 200 °C. Injector and detector temperatures were

200 °C and 220 °C, respectively. Retention time was 2.8 min. The minimum detection limit (MDL) for the method was 1.0 µg/sample.

The carbon disulfide samples were analyzed by Morse Laboratory in Sacramento, Calif., through the cooperation of Zeneca Ag Products, using NIOSH method 1600 with some modifications. The charcoal was extracted with 5.0 ml toluene that had been prechilled with dry ice over a period of at least 60 minutes and less than 3 hours. The limit of quantification was 1.0 ug/sample tube segment.

Sixteen additional air samples for method comparison were collected at sites 2, 4, 5, and 6, during intervals 1, 2, and 3, and sites 1, 4, and 5 during interval 4, on the same machine as the primary MITC sample. The samples were sent to Zeneca Ag Products, in Richmond, CA for analysis of MITC.

Method Validation

Before the field study, the CDFA Chemistry Laboratory Services determined the efficiency of the analytical method for analysis of MITC on the charcoal. Results of this determination are presented in Appendix C. Overall recoveries from the charcoal tubes ranged from 81 to 89%. Low recoveries (32 to 36%) of the spiked field samples were probably the results of a period of dry ice depletion that occurred in the ice chest containing the spikes during an extremely hot afternoon. However, the ice chest containing the samples was maintained with an adequate amount of dry ice. Spiked samples held in a -30° C freezer for the same time period resulted in recoveries of 75 to 77%.

RESULTS AND DISCUSSION

Weather data

A summary of the weather data for each sampling interval is listed in Table 2 .

Table 2. Summary of wind speed, direction, air temperature and relative humidity during MITC air monitoring.

Sampling Interval ¹	Wind Speed (mph)		Horiz. Wind Direction (°) Prevailing ²	Temperature (°C)		Rel. Humidity (%)	
	Range	Ave		Range	Ave	Range	Ave
1	1-10	6	190	27-35	31	23-45	33
2	3-7	5	90	25-27	26	34-51	40
3	0-10	6	55, 220 ⁺	21-29	26	24-61	37
4	0-8	5	320	27-37	33	18-40	29
5	3-11	6	320	29-39	36	14-38	22
6	1-11	7	225	23-31	27	25-62	41
7	3-10	7	320	29-36	33	20-51	32
8	0-11	7	235	20-31	25	25-56	37
9	1-9	6	320	17-48	29	27-37	33

¹Ambient air sampling for MITC (see Table 1).

²Direction in which the wind blew for most of the sampling interval .

⁺Predominant wind direction changed during sampling interval.

Soil Characteristics

The Soil Conservation Service (personal communication) describes the Cerini Loam soil of the treated area as a fine-loamy, mixed (calcareous), thermic Typic Torrifluent which is moderately alkaline (pH 7.9-8.4). Table 3 contains the results of characterization performed by mechanical analysis (Bouyoucos 1962, Hausenbuiller 1972, Calif Fertilizer

Assoc 1980) at the EHAP laboratory in Fresno, Calif., of soil samples collected at the north and south ends of the field.

Table 3. Soil characteristics of the application site.

Average Particle Size Distribution	
% sand	45
% silt	28
% clay	27
Textural Class (USDA)	loam
Soil pH	7.8
% Organic Carbon	1.18
Surface Irrigation water pH	7.9

MITC

Results of the MITC samples analyzed by the CDFA Chemistry Laboratory Services are presented in Table 4 and raw data in Appendix A. Stauffer Chemical Co. (method RRC-82-35, Leung 1982) stated that in conditions of low MITC concentrations and high relative humidity (above 80%), humidity had a serious negative effect on the recoveries. Stauffer determined that the use of the silica gel pre-tube at relative humidities above 80% resulted in no significant differences from recoveries at lower relative humidities. The MITC raw data (Appendix A), however, shows that during interval 2 when the silica pre-tube was used, results for the silica gel media indicated that it retained a high percentage (58-100%) of the total concentration of MITC passing through the sample set. Retention of MITC on the silica gel during interval 1 ranges from 0-4%. Concentrations for intervals 1 and 2 were calculated as a total of the sampling media and silica gel tube concentrations. There was no breakthrough of MITC to the backup section of the charcoal tubes in any

of the samples. The reason for the retention of MITC on the silica gel is not clear; although, silica gel is used as a trapping media for various organic substances.

Table 4. MITC air concentration (ppb).

Sampling Interval	Sampling Location									
	1 (5 m)	2 (5 m)	3 (5 m)	4 (5 m)	5 (75 m)	6 (75 m)	7 (75 m)	8 (75 m)	9 (150 m)	10 (150 m)
1	2450	2140	78.3	82.1	2110	44.2	4.71	23.9	1320	11.7
2	539	145	307	514	367	ND(13)	209	513	473	76.4
3	*	1050	177	178	548	35.7	24.1	44.2	49.7	12.7
4	10.1	46.0	106	12.2	ND(4)	6.51	6.64	ND(4)	ND(4)	4.69
5	47.1	239	147	ND(4)	47.4	42.3	ND(4)	ND(4)	ND(4)	6.50
6	116	130	8.16	ND(2)	133	2.27	5.68	2.29	8.06	ND(2)
7	7.81	18.3	20.0	ND(2)	5.75	7.85	ND(2)	ND(2)	*	ND(2)
8	6.22	16.4	ND(2)	ND(2)	14.5	ND(2)	ND(2)	ND(2)	*	ND(2)
9	ND(2)	2.28	2.29	ND(2)	ND(2)	ND(2)	ND(2)	ND(2)	*	ND(2)

* No sample

ND = None detected, the detection limit (ppb) shown in parenthesis

High concentrations of MITC during interval 1 indicate that metam-sodium was being rapidly degraded into MITC and volatilizing into the air during application (Table 4). Incorporation of metam-sodium in the soil and surface sealing to slow volatilization of MITC by subsequent application of irrigation water only, resulted in lower concentrations for the watering-in period (interval 2). During the next 6-hour interval (interval 3), high concentrations were again detected. The subsequent sampling intervals (4-9), resulted in decreasing concentrations over time, except for a slight increase during interval 5 where

the effect of high air temperatures (29-39 °C) may have caused an increase in volatilization. By interval 8 most samples were at a level that was below the detection limit.

The results from the study differ from concentrations of MITC reported by Rosenheck (1993) during an application of 2560 l (670 gals) of metam-sodium to a 2.7-ha (6.7 ac) field in May of 1992 in which the highest concentrations of MITC occurred 4 to 8 hours after sprinkler application of metam-sodium rather than during the application. At 4 to 8 hours after application the levels of MITC ranged from 1300 $\mu\text{g}/\text{m}^3$ (435 ppb) to 164 $\mu\text{g}/\text{m}^3$ (55 ppb). At 48 to 52 hours, concentrations ranged from 27 $\mu\text{g}/\text{m}^3$ (9.1 ppb) at 5 m to 5.5 $\mu\text{g}/\text{m}^3$ (1.8 ppb) at 500 m downwind from the field. The difference in results may be due to cooler temperatures or the smaller size of the application site in comparison to ours.

Since MITC concentration levels would naturally be higher downwind compared to upwind from the field, a graphic display of the meteorological data is helpful in analyzing the sample results. Wind roses (Wark and Warner 1981) created from the meteorological data illustrate the frequency distribution of wind direction as well as speed (Figure 2). The spokes represent the direction in which the wind is blowing, while the length is proportional to the duration. Each ring represents 10, 20, and 30 percent, respectively, of the time the wind was blowing in that direction. The different widths of each spoke represents a wind speed range.

During application (sampling interval 1), the highest concentrations of MITC were found 5 m downwind from the field edge. The air concentration 75 m downwind from the corner of the field was only slightly lower at 2110 ppb. At 150 m downwind from the field the MITC levels decreased by almost 50% to 1320 ppb, but were still high in comparison to the levels in the upwind direction. Concentrations during interval 2, when the water seal was being applied to the soil surface, dropped to 539 ppb 5 m downwind from the field edge. The highest levels found during interval 3 were also 5 m downwind from the field. The sample from site 1 during interval 3 was lost.

Figure 2. Wind roses and MITC concentration (ppb) for each sampling interval.

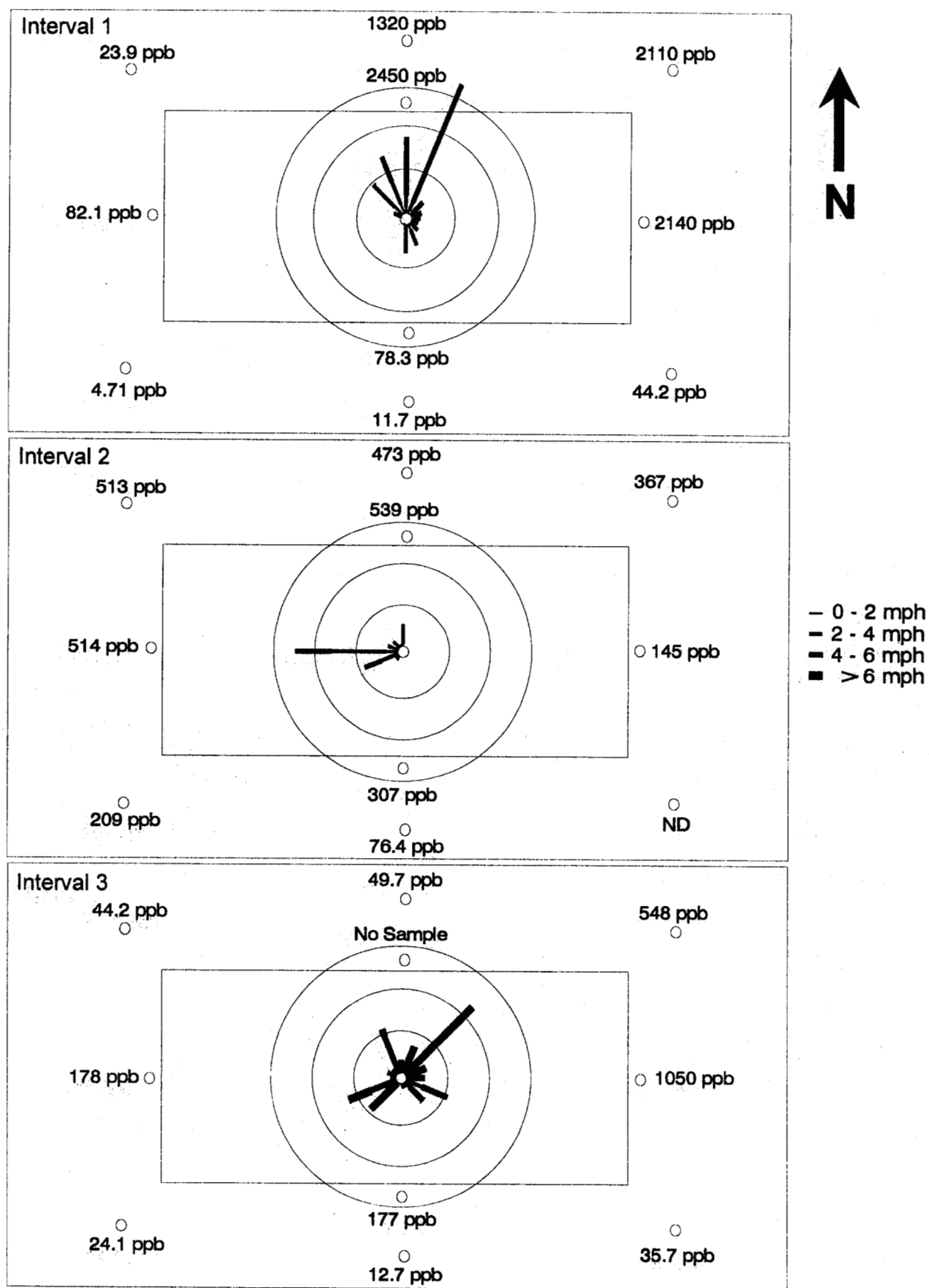


Figure 2. (continued)

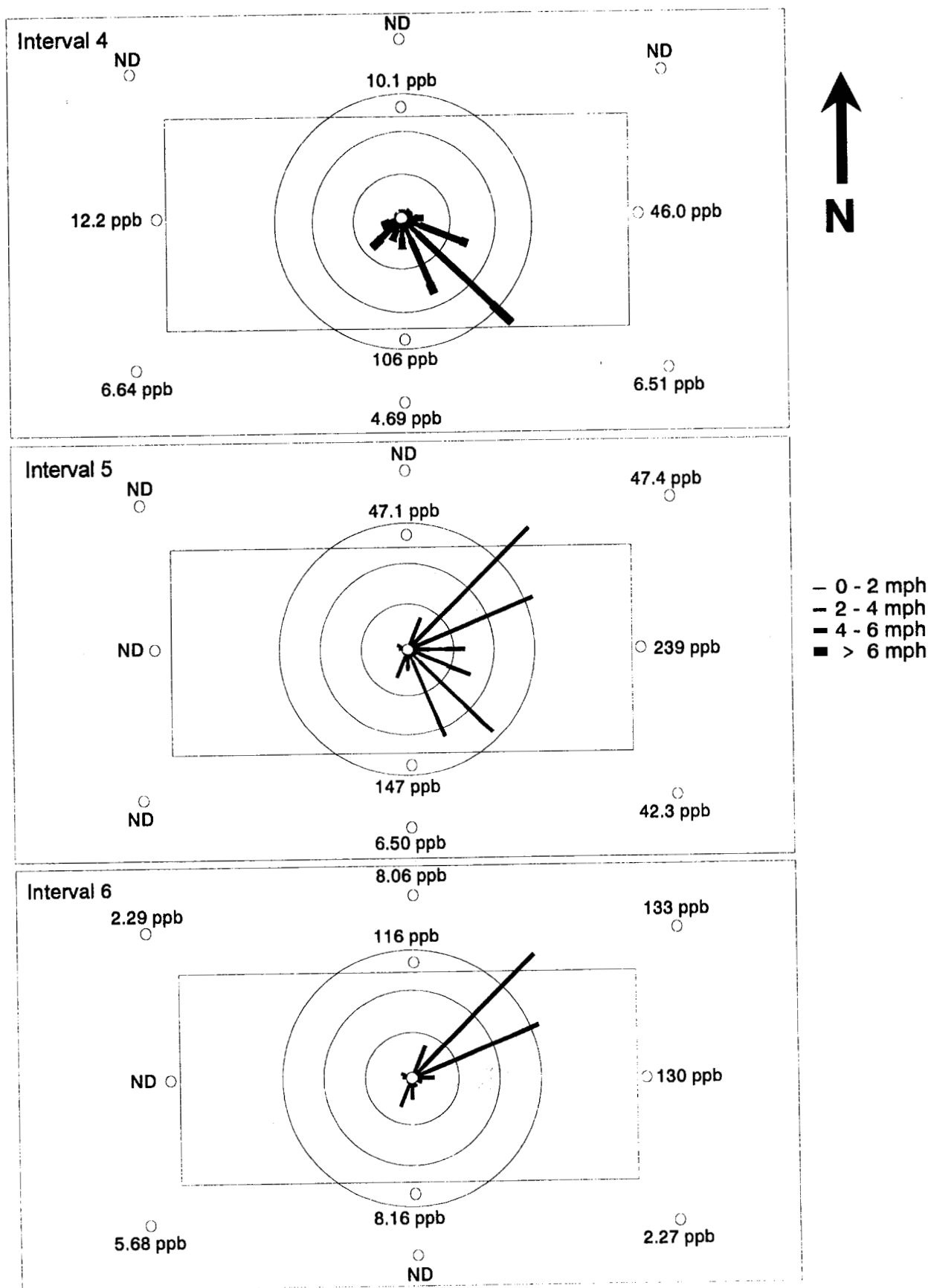
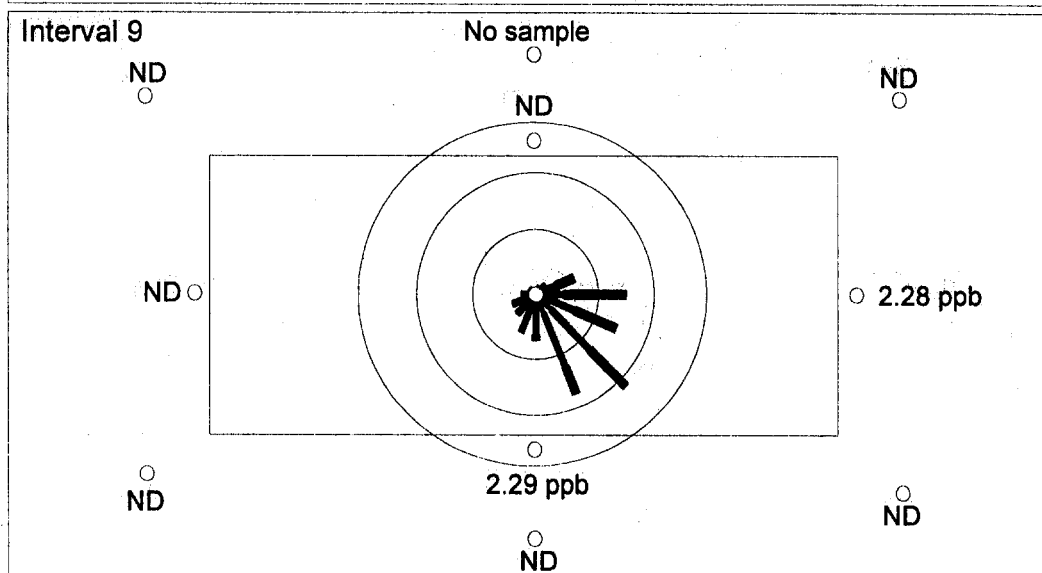
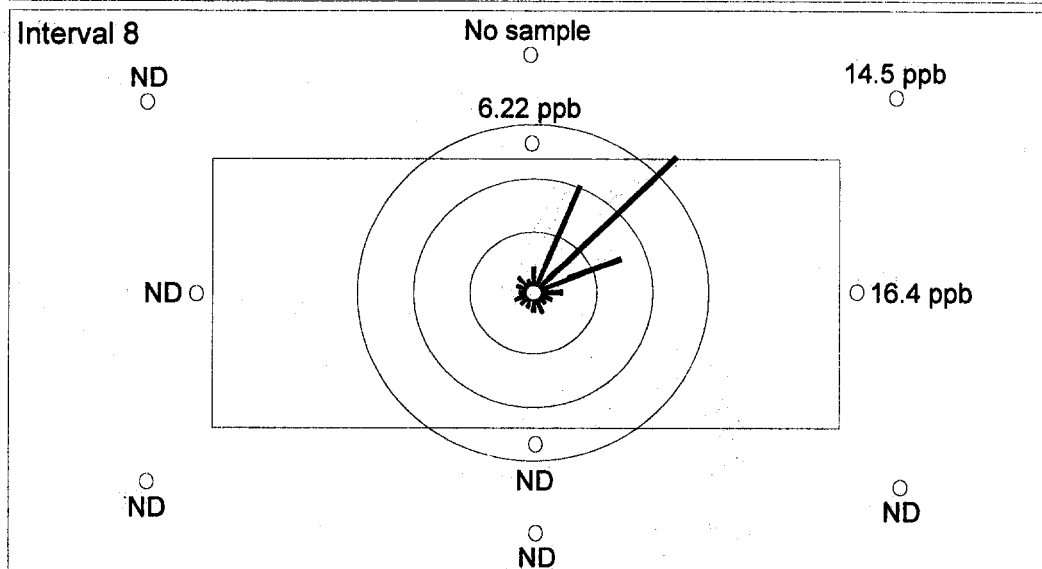
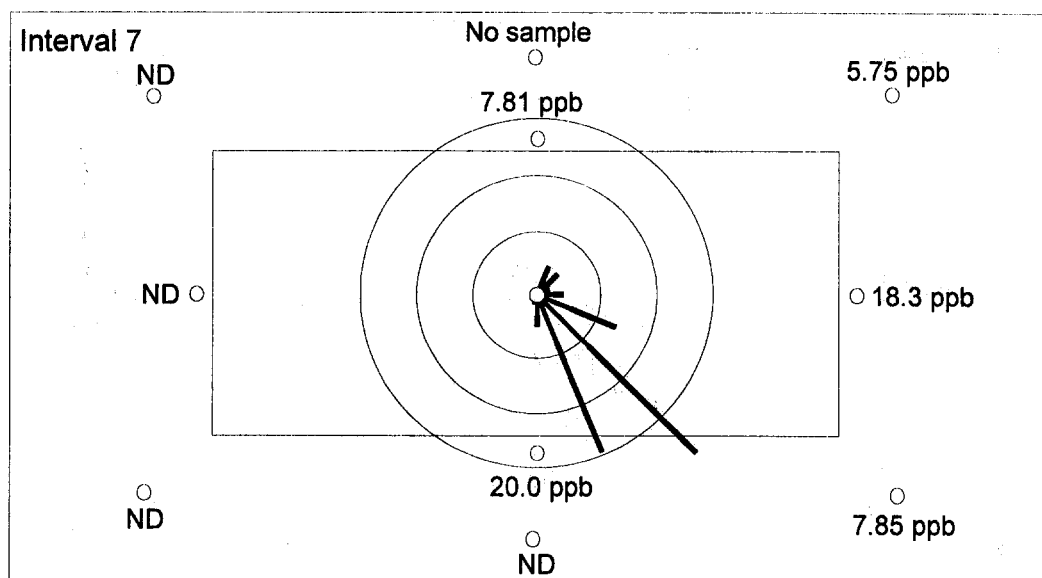


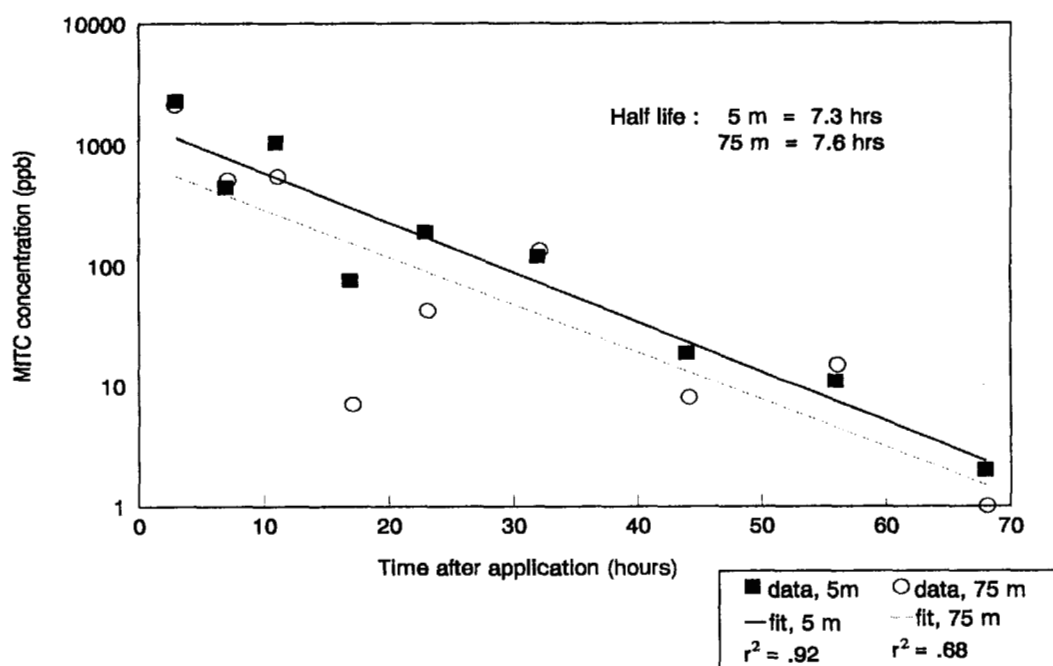
Figure 2. (continued)



- 0 - 2 mph
- 2 - 4 mph
- 4 - 6 mph
- > 6 mph

An approximation of the dissipation rate of MITC volatilized from the soil after chemigation can be determined by plotting log concentrations against time. Downwind concentrations from a single site or the average of multiple downwind sites were used to fit a regression line at 5 m and 75 m from the edge of the field (Figure 3). The half life for the dissipation of MITC was estimated at 7.3 to 7.6 hrs. A short half life (13 hrs) was also reported by Iwata (1989) for the dissipation rate of metam-sodium residue in soil from a field treated by chemigation.

Figure 3. MITC dissipation from field after chemigation of metam-sodium.



Hydrogen Sulfide (H₂S)

Levels of hydrogen sulfide (H₂S) above the detection limit of 3 ppb were detected up to 21 hours after the start of application (Table 5). Because H₂S is a minor breakdown product of metam-sodium, a relatively low concentration was expected to be present as the metam-sodium degraded. During application air concentrations fluctuated, so several

instantaneous readings were taken at each site to determine a concentration range. The highest level detected (76 ppb) occurred during application (interval 1) indicating that metam-sodium was being rapidly broken down and the H₂S released. No detectable levels were found during the watering-in period (interval 2) and following sampling times until the following afternoon (interval 5), at which time downwind levels ranged from 3 to 8 ppb. Subsequent monitoring 6 and 12 hours later failed to detect measurable concentrations. Raw data for H₂S is located in Appendix D.

H₂S concentrations measured during the study appear to be higher than levels reported in a previous study by the Air Resources Board (ARB 1994). Using the Jerome monitor shortly after a soil incorporated application, ARB measured 10 ppb of H₂S 3 inches directly above the soil. ARB reported less than 8 ppb of H₂S at 14 to 23 m downwind from the application site. Monitoring the air directly above a surface spill yielded a maximum of 50 ppb and concentration above an open metam-sodium tank ranged from 125-154 ppb (ARB 1994).

Table 5. Concentration readings (ppb) for hydrogen sulfide.

Sampling site	H ₂ S range (ppb)		
	Sample time after start of application		
	1 - 4 hrs	5 - 7 hrs	21 - 24 hrs
1	22 - 69	ND ^a	8
2	44 - 50	ND ^a	3
3	ND ^a	ND ^a	ND ^a
4	ND ^a	ND ^a	ND ^a
5	66 - 76	ND ^a	4
6	50 - 72	ND ^a	ND ^a
7	ND - 3	ND ^a	ND ^a
8	ND ^a	ND ^a	ND ^a
9	ND ^a	ND ^a	3
10	ND ^a	ND ^a	ND ^a

^aNone Detected, detection limit = 3 ppb

Carbon Disulfide (CS₂)

As a hydrolytic degradation product of metam-sodium in acidic conditions, carbon disulfide (CS₂) was not expected to be produced in high concentrations from the moderately alkaline soil and irrigation water (pH 7.9). Although CS₂ was detected in trace amounts in eight of the 16 samples (Appendix E) all were under the detection limit of 1.0 µg/segment (4 ppb). In a previous worker exposure study (Meyers 1993), CS₂ levels were below the laboratory minimum quantifiable limit of 1 µg during both sprinkler and soil injected applications and did not appear to play a significant role in worker exposure.

MITC Results Comparison

MITC results from the Zeneca Ag Products laboratory (Appendix B) for sampling intervals 1, 3, and 4 compare very well with those obtained from the CDFA Chemistry Laboratory Services. The Zeneca concentrations during interval 2 were considerably lower apparently due to the adsorption of the MITC onto the silica gel pre-tube which was not analyzed by Zeneca based on the understanding that the silica gel would not retain any significant MITC concentrations. Since MITC does adsorb to the silica gel, the necessity for the use of silica gel as a drying tube should be carefully determined depending on the conditions of the individual study.

CONCLUSIONS

MITC and H₂S, breakdown products of the soil fumigant metam-sodium, were detected in quantifiable concentrations in the air surrounding a fixed-set sprinkler application of metam-sodium. The highest concentrations of MITC occurred during application and immediately following the watering-in period. Concentrations during application ranged from 78.3 to 2450 ppb at 5 m from the field edge and 11.7 to 1320 ppb 150 m from the field, with the highest concentration measured in the downwind direction. Instantaneous H₂S readings were also highest during application, ranging from 3-76 ppb around the field.

All samples analyzed for CS₂ below the laboratory quantification limit of 1.0 µg/sample tube segment.

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Appendix A - MITC raw data for CDFA Chemistry Laboratory Services samples

Appendix A - MITC raw data for CDFA Chemistry Laboratory Services samples.

Interval	Sample No.	Media Type	Site	Flow (ml/min)	Time on	Time off	Run time (min)	ug/tube	RESULTS		
									Total	ug/L	ppb
BG	75	charc	north	295	837	1851	614	ND	ND	ND	ND
BG	301	charc	south	272	846	1845	598	ND	ND	ND	ND
1	240	silica	1	240	1926	113	347	9.69			
	77	charc	1	240			347	595.24	604.93	7.26	2450
	378	silica	2	262	1928	128	358	6.28			
	305	charc	2	262			358	590.99	597.27	6.37	2140
	251	silica	3	246	1928	112	344	ND			
	292	charc	3	246			344	19.69	19.69	0.23	78.3
	354	silica	4	247	1925	122	348	ND			
	361	charc	4	247			357	21.49	21.49	0.24	82.1
	376	silica	5	247	1941	155	376	1.25			
	310	charc	5	247			376	580.20	581.45	6.26	2110
	371	silica	6	252	1922	107	346	ND			
	293	charc	6	252			346	11.59	11.59	0.13	44.2
	407	silica	7	244	1925	110	343	ND			
	61	charc	7	244			343	1.17	1.17	0.01	4.71
	237	silica	8	250	1925	132	363	ND			
	280	charc	8	250			363	6.45	6.45	0.07	23.9
	380	silica	9	235	1926	117	351	14.54			
	289	charc	9	235			351	316.75	331.29	4.02	1320
	252	silica	10	240	1928	118	350	ND			
	291	charc	10	240			350	2.92	2.92	0.03	11.7
2	370	silica	1	250	113	241	88	20.54			
	309	charc	1	250			88	14.67	35.21	1.60	539
	229	silica	2	253	138	250	73	7.95			
	86	charc	2	253			73	ND	7.95	0.43	145
	400	silica	3	248	111	239	87	14.10			
	312	charc	3	248			87	5.55	19.65	0.91	307
	339	silica	4	255	122	241	74	24.13			
	335	charc	4	255			74	4.67	28.8	1.53	514
	391	silica	5	243	159	302	61	16.15			
	55	charc	5	243			61	ND	16.15	1.09	367
	379	silica	6	256	121	238	76	ND			
	83	charc	6	256			76	ND	ND	ND	ND
	397	silica	7	245	111	236	82	8.87			
	273	charc	7	245			82	3.58	12.45	0.62	209
	395	silica	8	247	153	252	58	21.83			
	290	charc	8	247			58	ND	21.83	1.52	513
	247	silica	9	244	124	256	90	21.83			
	308	charc	9	244			90	9.05	30.88	1.41	473

Appendix A. (continued)

Interval	Sample No.	Media Type	Site	Flow (ml/min)	Time on	Time off	Run time (min)	ug/tube	RESULTS		
									Total	ug/L	ppb
	401	silica	10	247	119	246	86	4.82			
	313	charc	10	247			86	0.00	4.82	0.23	76.4
3	57	charc	2	275	320	909	351	300.00		3.11	1050
	52	charc	3	274	243	856	373	53.64		0.52	177
	295	charc	4	290	245	911	383	58.71		0.53	178
	299	charc	4	265	245	912	382	25.98		0.26	86.4
	33	charc	5	265	310	922	373	160.98		1.63	548
	62	charc	5	258	306	922	376	164.77		1.70	572
	63	charc	6	273	246	850	364	10.55		0.11	35.7
	298	charc	7	270	237	907	388	7.50		0.07	24.1
	297	charc	8	275	251	924	390	14.08		0.13	44.2
	76	charc	9	265	247	940	403	15.76		0.15	49.7
	311	charc	9	267	246	933	405	123.01		1.14	383
	53	charc	10	266	247	902	374	3.75		0.04	12.7
4	279	charc	1	284	937	1444	305	2.58		0.03	10.1
	276	charc	2	264	915	1430	315	11.37		0.14	46.0
	87	charc	3	275	859	1431	330	23.33		0.31	106
	88	charc	3	277	859	1431	331	45.45		0.50	167
	92	charc	4	270	918	1453	333	3.25		0.04	12.2
	278	charc	5	260	927	1436	308	ND		ND	ND
	272	charc	6	267	853	1424	320	1.67		0.02	6.51
	275	charc	6	278	904	1424	329	1.83		0.02	6.81
	90	charc	7	300	908	1446	338	2.00		0.02	6.64
	91	charc	8	255	924	1500	335	ND		ND	ND
	270	charc	9	262	943	1452	308	ND		ND	ND
	93	charc	10	285	903	1438	335	1.33		0.01	4.69
5	188	charc	1	295	1447	2043	356	14.70		0.14	47.1
	210	charc	2	275	1432	2049	374	73.01		0.71	239
	267	charc	3	274	1433	2153	378	45.24		0.44	147
	281	charc	4	264	1455	2056	359	ND		ND	ND
	185	charc	5	282	1439	2056	377	14.96		0.14	47.4
	211	charc	6	292	1427	2040	372	13.64		0.13	42.3
	266	charc	7	290	1447	2048	359	ND		ND	ND
	24	charc	8	247	1502	2101	359	ND		ND	ND
	25	charc	8	274	1502	2102	359	ND		ND	ND
	186	charc	9	276	1454	2052	358	ND		ND	ND
	190	charc	9	292	1458	2052	353	ND		ND	ND

Appendix A. (continued)

Interval	Machine	Media	Site	Flow (ml/min)	Time on	Time off	Run time (min)	ug/tube	RESULTS		
	No.	Type							Total	ug/L	ppb
	81	charc	10	292	1438	2102	383	2.16		0.02	6.5
6	176	charc	1	292	2047	924	755	75.76		0.34	116
	268	charc	2	278	2053	947	775	82.89		0.38	130
	302	charc	3	276	2158	905	727	4.86		0.02	8.16
	222	charc	4	264	2059	919	739	ND		ND	ND
	15	charc	5	261	2112	940	747	76.97		0.39	133
	13	charc	6	273	2044	850	724	1.33		0.01	2.27
	220	charc	7	270	2050	912	742	3.38		0.02	5.68
	265	charc	8	262	2106	933	745	1.33		0.01	2.29
	29	charc	9	281	2056	550	532	3.58		0.02	8.06
	51	charc	10	273	2105	859	713	ND		ND	ND
	218	charc	10	272	2105	859	713	ND		ND	ND
7	269	charc	1	265	925	2049	682	2.58		0.02	7.81
	307	charc	2	262	949	2057	667	9.50		0.05	18.3
	94	charc	3	275	904	2105	718	11.73		0.06	20.0
	79	charc	4	260	919	2132	733	ND		ND	ND
	26	charc	5	305	942	2043	657	3.42		0.02	5.75
	80	charc	5	291	942	2039	656	3.12		0.02	5.50
	287	charc	6	273	851	2115	746	4.75		0.02	7.85
	187	charc	7	255	914	2128	733	ND		ND	ND
	82	charc	8	265	932	2139	725	ND		ND	ND
	282	charc	10	245	901	2108	728	ND		ND	ND
8	227	charc	1	277	2050	832	700	3.58		0.02	6.22
	215	charc	2	284	2056	837	700	9.69		0.05	16.4
	226	charc	3	290	2106	855	710	ND		ND	ND
	65	charc	4	285	2133	909	694	ND		ND	ND
	64	charc	5	260	2041	826	702	7.86		0.04	14.5
	37	charc	6	259	2119	941	682	ND		ND	ND
	44	charc	6	261	2119	941	682	ND		ND	ND
	177	charc	7	269	2128	906	696	ND		ND	ND
	212	charc	8	268	2140	914	695	ND		ND	ND
	221	charc	10	270	2109	851	701	ND		ND	ND
9	213	charc	1	280	832	1937	665	ND		ND	ND
	304	charc	2	258	838	1942	664	1.16		0.01	2.28
	27	charc	3	279	855	1955	658	1.25		0.01	2.29
	173	charc	4	265	909	2033	681	ND		ND	ND
	195	charc	4	272	909	2033	681	ND		ND	ND

Appendix A. (continued)

Interval	Sample	Media	Site	Flow	Time	Time	Run time	RESULTS	
	No.	Type		(ml/min)	on	off	(min)	ug/tube	ug/L ppb
	214	charc	5	275	825	1931	667	ND	ND ND
	303	charc	6	266	844	1946	662	ND	ND ND
	223	charc	7	250	905	2008	663	ND	ND ND
	191	charc	8	270	915	2040	683	ND	ND ND
	225	charc	10	270	851	1957	665	ND	ND ND

To take into account the amount of air sampled, the raw sample results were converted from µg/sample to ppb by the following calculations:

$$\frac{\text{sample results } (\mu\text{g}) \times 1000 \text{ } \ell / \text{m}^3}{\text{flow rate of sampler } (\ell / \text{min}) \times \text{run time (min)}} = \mu\text{g}/\text{m}^3$$

$$\mu\text{g}/\text{m}^3 \div 73.1 \text{ (molecular weight of MITC)} \div 40.7 \text{ (moles / m}^3 \text{ air)} \times 1000 = \text{ppb}$$

Appendix B - MITC raw data for Zeneca Ag Inc. samples

Appendix B. MITC raw data for Zeneca Ag Inc. samples

Sample No.	Interval	Machine No.	Site	Flow Rate (ml/min)	Adjusted Flow	Time on	Time off	Run time	ug/sample	RESULTS	
										ug/L	ppb
95	1	134	2	238	253	1928	130	358	450	7.47	2511.6
78	1	135	4	249	264	1925	125	348	28	0.43	143.1
96	1	74	5	233	248	1941	155	376	430	7.44	2500.9
294	1	277	6	237	252	1922	107	346	46	0.77	258.8
288	2	134	2	244	259	138	250	73	0.52	0.01	2.8
274	2	135	4	235	250	122	245	74	3.8	0.06	21.7
54	2	74	5	230	245	159	302	61	ND	ND	ND
85	2	277	6	235	250	121	238	76	ND	ND	ND
315	3	134	2	271	286	320	909	351	190	2.45	823.8
365	3	135	4	262	277	245	911	383	44	0.61	203.8
175	3	74	5	251	266	306	922	376	120	1.80	604.0
58	3	277	6	252	267	246	850	364	16	0.24	79.9
271	4	134	1	264	279	937	1444	305	2.5	0.03	11.4
89	4	135	4	259	274	918	1453	333	3.4	0.05	16.1
277	4	74	5	249	264	927	1436	308	ND	ND	ND

**Appendix C - MITC method validation and QA / QC
for CDFA Chemistry Laboratory Services samples**

Table 1. method validation data (% recovery) for the 1992 MITC air study.

Study: 124
 Chemical: MITC
 MDL: 0.2 ug/sample
 Date of Report: 10/02/

Matrix Sample Type: Coconut Base charcoal
 Lab: CDFA
 Chemist: Chow Hsiao

Lab sample #	Date Extracted	Date Analyzed	Results (ug)	Amount Added (ug)	Recovery %	X	SD	CV (%)
1183	9/30/92	9/30/92	3.84	5.0	76.9			
1183	9/30/92	9/30/92	4.01	5.0	80.2	79	2.3	3.0
1183	9/30/92	9/30/92	4.13	5.0	82.7			
1183	9/30/92	9/30/92	4.06	5.0	81.2	82	1.1	1.3
1183	9/30/92	9/30/92	4.16	5.0	83.2			
1184	9/30/92	9/30/92	84.00	100.0	84	84	0.6	0.7
1184	9/30/92	9/30/92	91.60	100.0	91.6			
1184	9/30/92	9/30/92	91.80	100.0	91.8	92	0.1	0.2
1184	9/30/92	9/30/92	88.60	100.0	88.6			
1184	9/30/92	9/30/92	87.50	100.0	87.5	88	0.8	0.9
1185	9/30/92	9/30/92	908.00	1000.0	90.8			
1185	9/30/92	9/30/92	849.00	1000.0	84.9	88	4.2	4.7
1185	9/30/92	9/30/92	889.00	1000.0	88.9			
1185	9/30/92	9/30/92	847.00	1000.0	84.7	87	3.0	3.4
1185	9/30/92	9/30/92	840.00	1000.0	84			
OVERALL:						85.4	4.38	5.13

X	SD	LWL	UWL	LCL	UCL
85	4.4	85	89	75.0	94.0

Table 2. Trapping Efficiency Study for the 1992 Air Monitoring Study (12 hour Sampling period).

Study: 124 Chemical: MITC MDL: 0.2 ug/sample Date of Report: 10/2/92				Matrix Sample Type: Coconut Base Charcoal Lab: CDFA Chemist: C. Hsiao					
Spike Level (ug)	Flow Rate	Glass Wool (ug)	Silica Gel (ug)	Coconut Charcoal		Recovery %	X	SD	CV (%)
				400 mg	200 mg				
5	1L/min	<0.2	<0.2	3.38	<0.2	68			
5	1L/min	<0.2	<0.2	3.31	<0.2	66	67	1.4	2.1
100	1L/min	<0.2	<0.2	75.7	<0.2	76			
100	1L/min	<0.2	<0.2	79.5	<0.2	80	78	2.8	3.6
1000	1L/min	<0.2	<0.2	788.8	<0.2	79			
1000	1L/min	<0.2	<0.2	822.2	<0.2	82	81	2.1	2.6
5	1L/min	<0.2	<0.2	3.2	<0.2	64			
5	1L/min	<0.2	<0.2	3.3	<0.2	66	65	1.4	2.2
100	1L/min	<0.2	<0.2	80.8	<0.2	81			
100	1L/min	<0.2	<0.2	80.2	<0.2	80	81	0.7	0.9
1000	1L/min	<0.2	<0.2	778.8	<0.2	78			
1000	1L/min	<0.2	<0.2	753.6	<0.2	75	77	2.1	2.8

Table 3. Continuing Quality Control Data (% Recovery) for the 1993 MITC Air Study.

Study: 124
 Chemical: MITC
 MDL: 1.0 ug/sample
 Date of Report: 8/23/93

Matrix Sample Type: Silica Gel
 Lab: CDFA
 Chemist J. Hernandez

Extraction Set No.'s	Results (ug)	Amount Spiked (ug)	Recovery (%)	X	SD	CV (%)
247,379,391,395,401	4.78	5.0	95			
397,370,229,400,339	4.75	5.0	95			
237,371,380,407,336,251, 240,252,378,354	3.85	5.0	77			
OVERALL:				89	10	12

Table 4. Continuing Quality Control Data (% Recovery) for the 1993 MITC Air Study.

Study: 124
 Chemical: MITC
 MDL: 1.0 ug/sample
 Date of Report: 8/23/93

Matrix Sample Type: Coconut Base Charcoal
 Lab: CDFA
 Chemist J. Hernandez

Extraction Set No.'s	Results (ug)	Amount Spiked (ug)	Recovery (%)	X	SD	CV (%)
55,83,290,308,313	4.78	5.0	95			
29,33,52,57,62,63,75,87,88, 92,270,276,279,295,298-9	3.92	5.0	78			
86,90,91,93,272,273,275,278, 309,312,325,1025-7	5.0	5.0	100			
24,25,81,190,211,266,185, 281,267,210,188,53,301, 311,76,297,186	4.31	5.0	86			
61,77,280,289,291,292,293, 305,310,361	4.24	5.0	85			
27,37,44,64,65,173,177,187, 191,195,212,215,223,225, 227,304,307,411	4.47	5.0	89			
26,79,80,82,94,214,221,269, 282,287,303,410,412	4.21	5.0	84			
213,265,218,15,302,222,176, 13,268,220,51,226	4.38	5.0	86			
OVERALL:				88	6.8	7.8

Appendix D - Raw Data for H₂S

Appendix D - Raw Data for H₂S

Sampling Site	Date	Start of Sampling Time	H ₂ S Concentration Range (ppb)
1	8/3/93	22:40*	69, 60 (wind change) 33, 24, 22
2		21:22*	44, 50
3		20:46*	ND
4		22:28*	ND
5		21:27*	74, 68, 66
6		21:14*	50, 50, 68
7		22:15*	3, ND
8		22:33*	ND
9		22:49*	ND
10		20:40*	ND
1	8/4/93	1:37	ND
2		1:19	ND
3		1:03	ND
4		2:02	ND
5		1:24	ND
6		1:09	ND
7		2:09	ND
8		1:56	ND
9		1:30	ND
10		0:59	ND
1	8/4/93	15:54	8
2		15:20	3
3		14:41	ND
4		16:16	ND
5		15:28	4
6		14:35	ND
7		16:11	ND
8		16:24	ND
9		15:50	3
10		14:47	ND

*readings at same spot within 5 min

Appendix E - CS₂ raw data from Morse Laboratory

Appendix E. CS₂ raw data from Morse Laboratory

DATE SAMPLE RECIEVED: 10/29/92, 8/16/93
 DATE SAMPLE EXTRACTED: 10/19/93
 DATE SAMPLE ANALYZED (INSTRUMENTATION): 10/21/93

SAMPLES STORED IN FREEZED AT -20 + 5 C
 ANALYTICAL METHOD: NIOSH 1600 w/modification 10-19-92
 CHEMICAL SPECIES ANALYZED FOR: Carbon disulfide
 LIMIT OF QUANTITATION: 1.0 ug/segment

RAW ANALYTICAL DATA

Sample	segment	Added		mL	mL	final	Dil.	uL	Peak	ng	seg.	ug/sample		%
		ug	ug/seg	Solvent	Aliquot	Vol.	Fact	Inj.	mm	Found	Inj.	found	corr.	Recovery
CHECK	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
Spike	front	1	1	5.0	3.0	3.0	1	2	23	0.275	4 x .001	0.688		69
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
Spike	front	50	50	5.0	3.0	3.0	20	2	166	0.875	2 x .0001	43.800		88
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
332	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
333	front	-	-	5.0	3.0	3.0	1	2	2	0.065	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
334	front	-	-	5.0	3.0	3.0	1	2	2	0.065	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
336	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
337	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
360	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
364	front	-	-	5.0	3.0	3.0	1	2	2.5	0.0725	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
369	front	-	-	5.0	3.0	3.0	1	2	2	0.065	4 x .001	0.000	<1.0	
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	
Reagent				5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0	

Appendix E. (continued)

DATE SAMPLE RECEIVED: 10/29/92, 8/16/93
 DATE SAMPLE EXTRACTED: 10/19/93
 DATE SAMPLE ANALYZED (INSTRUMENTATION): 10/21/93

SAMPLES STORED IN FREEZER AT -20 + 5 C
 ANALYTICAL METHOD: NIOSH 1600 w/modification 10-19-93
 CHEMICAL SPECIES ANALYZED FOR: Carbon disulfide
 LIMIT OF QUANTITATION: 1.0 ug/segment

Sample	segment	Added ug	Added ug/seg	mL Solvent	mL Allquot	final Vol.	Dil. Fact	uL Inject.	Peak mm	ng Found	seg. Inj.	ug/sample found corr.	% Recovery
CHECK	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
Spike A	front	1	1	5.0	3.0	3.0	1	2	22	0.255	4 x .001	0.638	64
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
Spike B	front	50	50	5.0	3.0	3.0	20	2	145	0.800	2 x .0001	40.000	80
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
224	front	-	-	5.0	3.0	3.0	1	2	1	0.038	4 x .001	0.095	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
296	front	-	-	5.0	3.0	3.0	1	2	1	0.038	4 x .001	0.095	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
300	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
314	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
324	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
325	front	-	-	5.0	3.0	3.0	1	2	3	0.075	4 x .001	0.188	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
326	front	-	-	5.0	3.0	3.0	1	2	1	0.038	4 x .001	0.095	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
328	front	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
	back	-	-	5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0
Reagent				5.0	3.0	3.0	1	2	0	0.000	4 x .001	0.000	<1.0